MORINGA OLEIFERA A POSSIBLE SOURCE OF BIODIESEL

SITI HAIZUM BT ABU HASSAN

Thesis submitted in partial fulfilment of the requirements
for the award of the degree of
Bachelor of Chemical Engineering (Gas Technology)

Faculty of Chemical & Natural Resources Engineering
UNIVERSITI MALAYSIA PAHANG

JULY 2013

ABSTRACT

Biodiesel is an alternative fuel, produced from domestic and renewable resources. It is biodegradable, non-toxic, and essentially free of sulphur and aromatics. Biodiesel is made through a chemical process called transesterification where triglycerides is working as a main component of vegetable oils, react with an alcohol then produce fatty mono-alkyl esters and glycerol. Currently, biodiesel appears to give the best renewable alternatives to petroleum-based diesel fuel, thus this research is to investigate the possibility of promoting *Moringa oleifera* as a potential source of biodiesel fuel. The objective of this research was to produce biodiesel from *Moringa oleifera* seeds, to determine the fatty acid composition and to check the physical properties of *Moringa oleifera* oils**.** The majority of biodiesel is produced via base-catalyzed transesterification with methanol. Transestrification of *Moringa oleifera* oil was carried out by a standard procedure employing a 6:1 molar ratio of methanol to vegetable oil using potassium hydroxide (KOH) as a catalyst. The agitation speed was set at 250 rpm for 90 minutes. Based on the result, around 60 ml of biodiesel was produced and tested. The *Moringa oleifera* methyl esters (MOME) is tested by GCMS to get the fatty acid percentage. For physical properties, the test showed the properties of biodiesel produced with density of 872.7 kg/m^3, *cetane* number of 64.75, cloud point at 10.7 °C, pour point at 6.1 °C and kinematic viscosity of 4.93 mm^2/s. The results showed that the properties are accepted within ASTM D6751 and EN 14214.

ABSTRAK

Biodiesel adalah bahan api alternatif, yang dihasilkan daripada sumber-sumber domestik dan boleh diperbaharui. Ia adalah mesra alam, tidak toksik, dan pada dasarnya bebas sulfur dan aromatik. Biodiesel adalah dibuat melalui proses kimia yang dipanggil transesterifikasi dimana trigliserida bekerja sebagai satu komponen utama minyak sayuran, bertindak balas dengan alkohol kemudian menghasilkan ester lemak mono-alkil dan gliserol. Pada masa ini, biodiesel muncul untuk memberikan alternatif terbaik yang boleh diperbaharui kepada bahan api diesel berasaskan petroleum, dengan itu kajian ini adalah untuk menyiasat kemungkinan menggalakkan *Moringa oleifera* sebagai sumber berpotensi bahan api biodiesel. Objektif kajian ini adalah untuk menghasilkan biodiesel daripada benih *Moringa oleifera* dan untuk menentukan komposisi asid lemak dan untuk memeriksa sifat-sifat fizikal minyak *Moringa oleifera*. Majoriti biodiesel dihasilkan melalui transesterifikasi asas-pemangkin dengan metanol. Transestrifikasi minyak *Moringa oleifera* telah dijalankan oleh satu prosedur standard yang menggunakan nisbah 6:1 molar metanol dengan minyak sayur-sayuran menggunakan kalium hidroksida (KOH) sebagai pemangkin. Kelajuan pergolakan telah ditetapkan pada 250 rpm selama 90 minit. Berdasarkan keputusan itu, kira-kira 60 ml biodiesel dihasilkan dan diuji. *Moringa oleifera* metil ester (MOME) diuji dengan GCMS untuk mendapatkan asid peratusan lemak. Untuk ciri-ciri fizikal, ujian menunjukkan sifat-sifat biodiesel dihasilkan dengan ketumpatan 872.7 kg/m^3, bilangan setana 64.75, takat beku pada 10.7 ° C, takat cair pada 6.1 ° C dan kelikatan kinematik 4.93 mm^2 / s. Hasil kajian menunjukkan bahawa sifat-sifat yang diterima dalam ASTM D6751 dan EN 14214.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

LIST OF ABBREVIATIONS

ASTM	American Society of Testing and Materials
CH_3ONa	Sodium Methoxide
CN	Cetane Number
EI	Electron impact
EN	Europen Standard
EtOH	Ethanol
FFA	Free Fatty Acid
FAME	Fatty Acid Methyl Ester
FAMEs	Fatty Acid Methyl Esters
GC	Gas Chromatography
Kg	Kilogram
KOH	Potassium Hydroxide
MO	*Moringa oleifera*
MOME	*Moringa oleifera* methyl ester
MeOH	Methanol
MSD	Mass selective detector
NaOH	Sodium Hydroxide
UMP	University Malaysia Pahang

1 INTRODUCTION

1.1 Motivation and problem statement

The worldwide resource of coal, natural gas, and most importantly oil are depleting very fast. Without alternative energy sources, the world will experience a severe lack of energy in the future. It is vital to discover alternative energy sources in order to secure industrial and commercial future. Hence, biodiesel holds a great potential as alternative energy source. Biodiesel is the name of a clean burning alternative fuel, produced from domestic, renewable resources. Biodiesel is usually made from plant oils or animal fat through a series of chemical reactions. It is safe and contains no petroleum, but it can be blended at any level with petroleum diesel to create a biodiesel blend. It can be used in compression-ignition (diesel) engines (Knothe et al., 2005; Mittlebach & Remschmidt, 2004) with little or no modifications. Biodiesel has better properties than petroleum diesel properties such as; biodegradable, safe to use (nontoxic), and essentially free of sulfur and aromatics. (Martini & Shell, 1998) stated that biodiesel fuel has the potential to reduce the level of pollutants and the level of potential or probable carcinogens. Oilseed crops such as soybean, canola and sunflower are particularly useful for producing biodiesel, (Peterson, 1991; Mittlebach, 1990) stated that production of biodiesel from conventional sources (palm, sunflower, & soybean) increasingly has placed strain on food production, availability and price . Other new examples, studies of biodiesel from less common or unconventional oils include *Moringa oleifera* (Rashid et al., 2011), tobacco (Usta, 2005), Pongamia (Karmee & Chadha, 2005), Jatropha (Foidl et al., 1996) and rubber seed oils (Ikwuagwu et al., 2000; Ramadhas et al., 2005). In order to take advantage of potential international opportunities, research for an additional regional biodiesel feedstock should be investigated. Therefore, this study will be focused on the *Moringa oleifera* as a possible source of biodiesel.

Moringaceae is as single-genus family of oilseed trees with 14 unknown species (Rashid et al., 2008), all of which are growing in tropical and sub-tropical climates. Figure 1.1 shows the most popular species is *Moringa oleifera*, a multi-purpose tree; that has a lot of different uses and benefits which all parts are edible and can be used for oil, fibre, medicine, and water purification. The leaves, fruits, flowers, and immature

pods of this tree are edible and they form a part of traditional diets in many countries of the tropics and sub-tropics (Siddhuraju & Becker, 2003; Anhwange et al., 2004). *Moringa oleifera*, indigenous to sub-Himalayan regions of northwest India, Africa, Arabia, South-east Asia, the Pacific and Caribbean Islands and South Africa, is now distributed in the Philippines, Cambodia, and Central and North America (Morton, 1991). In Malaysia, there are several generic names that refer to *Moringa oleifera* like 'kachang kelur', 'lemunggai', 'meringgai', 'semunggai', 'smunggai', 'murunggai', 'murunggi' and 'remunggai'. (Sutherland et al., 1994) stated that the seeds of *Moringa oleifera* are considered to be antipyretic, acrid and bitter. *Moringa* seed kernels contain oil that is commercially known as "Ben oil" or "Behen oil". (Ndabigengeser & Narasiah, 1998) reported that, the ben oil was erroneously to be resistant to rancidity and used extensively in the 'enfleurage' process.

Figure 1-1: *Moringa oleifera* tree.

Nowadays, fossil fuels have become an integral part of our daily lives. They fuel transportation, industrial machinery and electricity. Therefore, our world now is confronted with two crises of fossil fuel depletion and environment degradation. (Singh & Singh, 2009) stated that the indiscriminate extraction and consumption of fossil fuels have led to a reduction in petroleum reserves. Historically, fossil fuels were available in abundant supply that was easy to obtain and transport. But now signals are given that the supply is running out and that it will take centuries to be restocked. Since it takes millions of years to create fossil fuels, demand for these products is raising rapidly (Kafuku & Mbarawa, 2010) which results in high diesel prices as well as adding to other negative effects of burning petroleum, such as air pollution and global warming.

There are plenty of solutions have been developed as scientists and researchers begin to realize the effect of fossil fuels on the natural world and economic environment.

(Kibazohi & Sangwan, 2011) reported that, to overcome the challenges of depleting fossil fuel resources and the threats from global warming and climate change, the world must look for alternatives. Currently, biodiesel appears to give the best renewable alternatives to petroleum-based diesel fuel, thus this research is to investigate the possibility of *Moringa oleifera* as a potential source of biodiesel fuel.

1.2 Objectives

The main objective of this research work is to evaluate *Moringa oleifera* seed oil as a potential raw material for biodiesel production and the specific objectives are to:

- To extract the oil content of *Moringa oleifera* seeds.
- To determine the fatty acid composition and physical properties of *Moringa oleifera* methyl ester oil.
- To synthesize *Moringa oleifera* seed oil esters using alkaline transesterification method.

1.3 Scope of this research

The scope will be limited to the study of the fatty acid composition of *Moringa oleifera* seeds oil. In other words, to investigate its chemical composition and also to know the physical properties of the oil. It will be carried out to prove that *Moringa oleifera* seed oil as a potential raw material for biodiesel production.

2 LITERATURE REVIEW

2.1 Overview

This paper presents a review about what is biodiesel and some of the standards that is used to compare the results. A brief explanation about *moringa* tree which known as miracle tree and also about transesterification process which is an important process to convert the pure vegetable oil to biodiesel, all will be discussed in this chapter.

2.2 Introduction

Biodiesel production is a very modern for researchers due to the relevance that it is winning everyday because of the increase in the petroleum price and the environmental damage (Marchetti et al., 2005). The name of biodiesel was introduced in the United States (US) during 1992 by the National Soy diesel Development Board which has pioneered the commercialization of biodiesel in the United States (Singh & Singh, 2009). As reported by US Standard Specification for Biodiesel (ASTM D6751), biodiesel is defined as a fuel comprised of mono-alkyl esters of long chain fatty acids derived from vegetable oils or animal fats. Some others biodiesel standards are ASTM D6751 (ASTM D American Society for Testing and Materials) and the European standard EN 14214, which was developed from previously existing standards in individual European countries (Knothe, 2005). Biodiesel can be made from methyl, ethyl, isopropyl, and other alcohols, but most biodiesel research focuses on methyl esters. (McCormick & Alleman, 2005) stated that, biodiesel has a lower emission profile than petro-diesel and decrease the greenhouse gas emission from combustion ignition engines; it is also an ecologically friendly fuel. Based on (Schuchardt et al., 1998; Knothe, 2005) studies, biodiesel contains little or no sulfur or carcinogenic polyaromatic components, easy to handle (flash point above 110 °C), and decreases soot emission. In addition, biodiesel fuel can generally be used in existing oil heating systems and diesel engines without modification (Knothe et al., 2005; Mittlebach & Remschmidt, 2004) and it can be distributed through existing diesel fuel pumps. This is an advantage over other alternative fuels, which can be expensive to use initially due to high cost of equipment modifications or new purchases.

Based on the chemical point of view, oils from different sources have a different percentage of fatty acid compositions. Because of different fatty acids have different physical and chemical properties; the fatty acid profile is probably the most crucial parameter influencing the corresponding properties of vegetable oil or animal fat. Chemically, the oil/fats consist of 90 - 98 % triglycerides and small amount of mono and disglycerides (Singh & Singh, 2009). These fatty acids differ in the length of carbon chains, orientation and position of double bonds in these chains. In most fats, the length of the fatty acid carbon chain ranges between C16 and C18. There are also differences in the degree of saturation (number and position of double bonds) in acid molecules. Saturation is the major factor determining physical properties of fats. Highly unsaturated vegetable oils are low viscosity liquids, while fully saturated animal fats are solid at ambient temperature. From the point of view of the transesterification process itself, these differences in molecular structure are insignificant in terms of process parameters or energy demand. Table 2.1 shows the fatty acid of *Moringa oleifera* with other vegetable oils.

Table 1-1: Free fatty acid (FFA) profile of *Moringa oleifera* with other vegetable oils (Rashid et al., 2008)

Fatty acid	Moringa oleifera	Palm[a]	Rapeseed[a]	Soybean[a]	Sunflower
C16: 0	6.5	44.1	3.6	11	6.4
C18: 0	6.0	4.4	1.5	4	4.5
C18: 1	72.2	39.0	61.6	23.4	24.9
C12: 2	1.0	10.6	21.7	53.2	63.8
C18: 3	-[b]	0.3	9.6	7.8	-[b]
C20: 0	4.0	0.2	-	-	-
C20: 1[c]	2.0	-	1.4	-	-
C22: 0	7.1	-	-	-	-
Other	1	1.1% C14: 0 traces of others	0.2% C22: 1	Traces	Traces

[a] Data from Gunstone and Harwood, 2007. These values constitute averages of numerous samples
[b] This may indicate traces (< 1%) or absence of these fatty acids.
[c]Eicosenoic acid.

2.3 Transesterification Process of Biodiesel

Biodiesel can be produced straight away from vegetable oil, animal oil fats. Common vegetable oils or animal fats are esters of saturated and unsaturated monocarboxylic acids with the trihydric alcohol glycerides (Leung et al., 2010), these esters are called tryglicerides which can react with alcohol with a presence of catalyst and the process is

known as transesterification. There are three basic methods to biodiesel production from oils and fats which are:

a) base catalyzed transesterification of the oil.
b) direct acid catalyzed transesterification of the oil.
c) conversion of the oil to its fatty acids and then to biodiesel.

Almost all biodiesel is produced using base catalyzed transesterification as it is the most economical process requiring only low temperatures and pressures and producing a 98% conversion yield. Transesterification also known as alcoholysis, where there is a displacement of alcohol from ester by another alcohol in a process similar to hydrolysis, except that alcohol is used instead of water (Srivastava & Prasad, 2000). The general reaction between tryglicerides and methanol is shown in Figure 2.1.

Vegetable oil + Methanol → Biodiesel + Glycerol

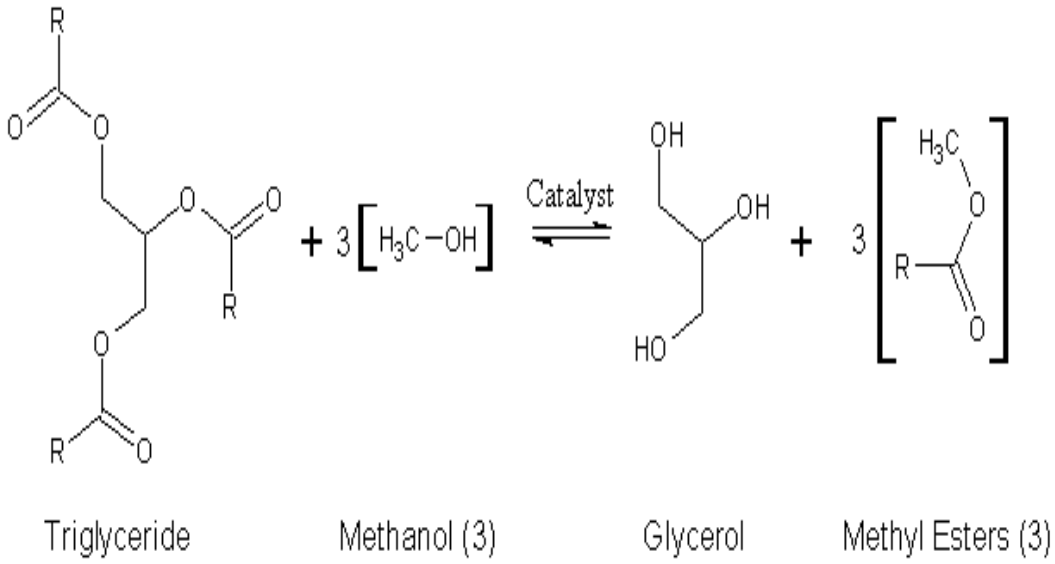

Figure 2-1: Transesterification reaction of vegetable oil with alcohol to esters and glycerol (Freedman, 1986).

Based on the reaction, triglycerides is working as a main component of vegetable oils, react with an alcohol then produce fatty mono-alkyl esters and glycerol. (Sprules & Price, 1950) stated that, alcohols are primary and secondary monohydric aliphatic alcohols having 1-8 carbon atoms. The alcohols that can be used in the transesterification process are; methanol, ethanol, propanol and butanol. In industry, nowadays methanol and ethanol are used most frequently in production, especially

methanol because of its low cost and its physical and chemical advantages as methanol is the shortest chain alcohol and polar. Ma &Hanna, (1999) reported that, methanol can react with triglycerides quickly and the alkali catalyst is easily dissolved in it. However, due to its boiling point (°C), there is a large explosion risk associated with methanol vapors which are colorless and odorless (Leung et al., 2010). Table 2.2 shows the advantages and disadvantages at different type of catalyst used in the biodiesel production.

Table 2-2: Advantages and disadvantages at different type of catalyst used in the biodiesel production. (Leung et al., 2010)

Type	Example	Advantages	Disadvantages
Alkali Homogeneus	Naoh, KOH	High catalytic activity, low cost, favorable kinetics, modest operation conditions	Low FFA requirement, anhydrous conditions, saponification, emulsion formation, more wastewater from purification, disposable
Heterogenous	CaO,$CaTiO_3$,$CaZrO_3$,CaO-CeO_2,$CaMnO_3$,$Ca_2Fe_2O_5$, KOH/Al_2O_3,KOH/NaY, Al_2O_3/Kl,ETS-10 zeolites, alumina/silica supported K_2CO_3	Noncorrosive environmentally benign, recyclable, fewer disposal problems, easily separations, higher selectivity, longer catalyst lifetimes	Low FFA requirement, anhydrous conditions, high molar ratio of alcohol to oil requirement, high reaction temperature and pressure, diffusion limitations, high cost
Acid Homogenous	Concentrated sulphuric acid	Catalyzed esterification and transesterification simultaneously, avoid soap formation	Equipment corrosion, more waste from neutralization, difficult to recycle, higher reaction temperature, long reaction times, weak catalytic activity
Heterogeneous	ZnO/I_2, ZrO_2/SO_4^{-2}, TiO_2/ SO_4^{-2}, carbon-based solid acid catalyst, Vanadyl phosphate, niobic acid, sulphate zirconia, Amberlyst-15, Naflon-NR50	Catalyzed esterification and transesterification simultaneously, recyclable, eco-friendly	Low acid site concentrations, low microporosity, diffusion limitations, high cost
Enzymes	Casnidaantartica fraction B lipase, Rhizomucormieher lipase	Avoid soap formation, non polluting, easier purification	Expensive, denaturation

2.4 Transesterification using alkali catalyst

By using alkali catalyst, the reaction is catalysed by a basic catalyst. A base catalyst is a chemical with a pH value greater than 7, it has the ability to give extra electrons. Sodium hydroxide (NaOH), potassium hydroxide (KOH), and sodium methoxide (CH_3ONa) are the most common homogeneous base catalysts employed during alkaline transesterification (Helwani et al., 2009; Demirbas., 2009). However, potassium hydroxides have been found to be more suitable for the ethyl ester biodiesel production, due to their high yields (Predojevic, 2008). The base catalysed process is the most commonly used because it can be performed at low temperature and pressure and yields high conversion (98%) within a short time. (Akoh et al., 2007) stated that, most of the base catalysed reactions were carried out at temperatures close to the alcohol boiling point (65 °C) and molar ratio of alcohol to oil of 6:1. Table 2.3 below shows the effect of the catalyst on the biodiesel purity and yield.

Table 2-3: Effect of the catalyst on the biodiesel purity and yield. (Vicente et al., 2004)

Catalyst				
	Sodium hydroxide	Pottasium hydroxide	Sodium methoxide	Pottasium methoxide
Biodiesel purity (wt%)	99.7	99.69	99.7	99.4
	99.75	99.8	99.69	99.5
	99.72	99.8	99.72	99.65
	99.65	99.74	99.75	99.53
	99.71±0.04	99.76±0.05	99.72±0.03	99.52±0.1
Biodiesel yield (wt%)	86.33	91.67	99.17	98.33
	86.67	91.67	99.33	98.5
	87	91.33	99.83	98.33
	86.71	92	99	98.67
	86.71±0.28	91.67±0.27	99.33±0.36	98.46±0.16

Temperature = 65°C, molar ratio = 6, catalyst = 1%

2.5 The miracle tree of Moringa oleifera

Moringa oleifera belongs to family *Moringaceae* that is represented only by a single genus (*Moringa)* with 14 unknown species and it is popularly known as 'The Miracle

Tree‘. The *Moringa* or Ben-oil tree is believed to be native to India, Arabia, and possibly even across Africa and the Caribbean. *Moringa oleifera* is a very fast growing tree (Hsu et al., 2006); it commonly reaches four meters in height just 10 months after the seed is planted and can bear fruit within its first year. Its pods are triangular in cross-section (30 to 50 cm long) and legume-like in appearance. These pods have oil rich black and winged seeds, which can be crushed to produce biodiesel. Oil qualities are similar to that of olive oil, and the degummed oil has favorable characteristics for use as a replacement for petroleum diesel. (Tsaknis et al., 1999; Rashid et al., 2008) stated that the biodiesel produced from *moringa* oil exhibit a high cetane number (CN) of approximately 67, one of the highest found for a biodiesel fuel, enhanced oxidative stability and high cloud point. In some parts of the world *Moringa oleifera* is referred to as the ‘drumstick tree’ or the ‘horse radish tree’, whereas in others it is known as the kelor tree (Anwar & Bhanger, 2003). There are many local names in malay for this tree like ‘kachang kelur’, ‘lemunggai’, ‘meringgai’, ‘semunggai’, ‘smunggai’, ‘remunggai’ and all of it refers to *Moringa oleifera* (*Moringa oleifera*, 2012).

2.6 Uses of Moringa oleifera

Moringa oleifera is the greatest plant yet discovered with a rich of nutrient. This miracle plant has been making strides in less-developed societies for thousands of years, and significant nutritional research has been conducted since the 1970s (Mahmood et al., 2010). *Moringa* provides a rich and rare combination of nutrients, amino acids, antioxidants, anti-aging and anti-inflammatory properties used for nutrition and healing. *Moringa* is sometimes called "Mother's Best Friend" and "Miracle Tree." (Siddhuraju & Becker, 2003; Anhwange & Ajibola, 2004) stated that the leaves, fruits, flowers, and immature pods of this tree are edible and they form a part of traditional diets in many countries of the tropics and sub-tropics. Ibrahim et al., (1974) has reported, *Moringa* seeds oil content and its properties show a wide variation depending mainly on the species and environmental condition. *Moringa oleifera* has many medicinal uses and has significant nutritional value as presented by Anwar et al., (2007).

3 MATERIALS AND METHODS

3.1 Introduction

In this chapter, the detailed description of the materials, equipments and methods are discussed.

a) Material
   - Raw material
   - Alcohol selection
   - Catalyst selection

b) Equipment
   - Hot plate with stirrer
   - 2 L round-bottomed flask equipped with a reflux condenser and thermometer
   - Separating funnel
   - Rotary evaporator
   - Gas chromatography
   - Viscosity equipment (Cole-Parmer)

c) Research method
   - Collecting sample
   - Experimented work
   - Product analysis

3.2 Materials

- **Raw Material**

The raw material used in this research was mature *Moringa oleifera* seeds collected from neighbourhood gardens around Banting, Selangor.

- **Alcohol selection**

Methanol (MeOH) and ethanol (EtOH) were utilized most frequently, especially methanol because of its low cost and its physical and chemical advantages. Therefore, it was used in the research experiment.

- **Catalyst selection**

A catalyst is required to facilitate the reaction between the alcohol and the oil. The most common catalysts used were potassium hydroxide, (KOH) and sodium hydroxide, (NaOH). In completing this experiment, potassium hydroxide was chosen as it is easy to handle and dissolve faster in methanol.

3.3 Methods

a) Collecting sample

It was collected from neighbourhood gardens around Banting, Selangor and surrounding areas where the plant was found growing in abundance.

b) Preparation of sample

Two kg of *Moringa oleifera* seeds was prepared and grinded to make it powder. Then, the crushed seeds were dried in the oven at 70 °C to remove the moisture content. In the laboratory, three sets of 6:1 ratios of methanol to oil were prepared.

c) Experiment

- **Extraction of *Moringa oleifera* (MO) seeds**

Weight the crushed MO of 10 g and placed in the thimble. The thimble was placed into the thimble holder of the soxhlet extractor and it was fitted with 2 L round-bottomed flask. Around 200 mL of n-hexane was poured into the bottom flask at 60 °C within three cycles each for 30 minutes. Then, the solvent was removed under vacuum using a rotary evaporator at 60 °C to obtain crude *Moringa oleifera* oil.

- **Transesterification of *Moringa oleifera* oil**

In order to have a 1.0 % concentration of KOH for every 50 mL of oil used, 0.5 g of potassium hydroxide (KOH) pellets was added in the methanol to produce methoxide

solutions. These solutions were prepared in a volumetric flask. The flask was tipped back and forth until KOH was dissolved.

- **Reaction of methoxide with *Moringa oleifera* oil**

A sample of 16 ml *Moringa oleifera* oil was poured into the 2 L round-bottomed flask. (Lang et al., 2001) reported that a molar ratio of methanol to oil of 6:1 was used in order to utilize 100% stoichiometric excess in methanol. Hence, the methoxide was carefully poured into the oil according to the ratio. Stirring speed was set up at 250 rpm as it will increase the intact area between oils and potassium hydroxide methanol solution. The reaction will only occurred only at interface of the two layers and considered too slow to be feasible. (Rashid et al., 2008)

- **Phase separation**

Once the transesterification was completed, the hot mixture from the 2 L round-bottomed flask was poured carefully into the separator funnel for settling for at least 12 hours. Two distinct liquid phases were formed during separation process. The crude ester phase was at the top, while the glycerol phase at the bottom. The bottom glycerol phase was removed and the crude *Moringa oleifera* methyl esters layer at the top was then washed with warm deionized water.

- **Washing methyl ester**

The *Moringa oleifera* methyl ester (MOME) produced from the reaction was washed with hot water. The water was poured in gently and carefully to get the product biodiesel.

3.4 Product analysis

To achieve the objective of the experiment, the fatty acid methyl esters (FAMEs) contents were determined by gas chromatography, model GC−6890N coupled with mass spectrometer, model MS−5973 MSD (mass selective detector). Separation was performed on a capillary column DB-5MS (30 m ×0.32 mm, 0.25µm of film thickness). The carrier gas was helium with flow rate of 1.5 mL/min. The column temperature was programmed from 120-300 °C at the rate of 10 °C/min. A sample volume of 1.0µL was

injected using a split mode, with the split ratio of 1:10. The mass spectrometer was set to scan in the range of m/z 50-550 with electron impact (EI) mode of ionization.

For physical properties, the biodiesel was prepared with a volume of 60 mL to run the biodiesel tests. The following testing was:

- Density at 15 °C
- Kinematic viscosity at 40 °C (mm^2/s)
- Cloud point (°C)
- Pour point (°C)
- *Cetane* number

3.5 Summary

In this chapter the steps of preparing the samples to produce biodiesel by using optimum ratio of methanol:oil, kinematic viscosity are discussed and the fatty acid profile of the biodiesel produced were explained.

4 RESULTS

4.1 Introduction

This chapter presents all the results obtained from the transesterification process using methanol:oil ratio of 6:1 and following the method described in Chapter 3. The test and analysis is done according to the objectives of this research. The main aim is to determine the fatty acid profile and the physical properties of the biodiesel produced from the *Moringa oleifera* methyl ester (MOME).

4.2 Biodiesel Yield

In this study, the parameters such as reaction time, reaction temperature and methanol:oil ratio were set up for 90 minutes, at 60 °C and 6:1 methanol to oil ratio respectively. The agitation speed was set up at 250 rpm.

4.3 Fatty acid composition of Moringa oleifera methyl ester

Table 4.1 shows the fatty ester profile of the *Moringa oleifera* oil as determined by GC. According to the results obtained, it agrees with the previous literature on *Moringa oleifera* oil (Anwar & Bhanger., 2003; Anwar et al., 2007). For the comparison purposes, Table 4.2 shows the fatty acids profile of palm, rapeseed (canola), soybean and sunflower oils compared with this research results and the results from (Rashid et al., 2008) in Table 1.1. The predominant acid in *Moringa* oils is oleic acid, 72.67 %. (Knothe et. al., 2005) reported that oils with high oleic acid content are being developed which would give biodiesel fuels with a reasonable balance of fuel properties.

Table 4-1 Fatty acid profile of *Moringa oleifera* seed oil

Fatty acid	Systemic name	Formula	Structure	Amount (wt %)
Palmatic	Hexadecanoic	$C_{16}H_{32}O_2$	16:0	7.59
Oleic	cis-9-Octadecenoic	$C_{18}H_{34}O_2$	18:1	72.67
Stearic	Octadecanoic	$C_{18}H_{36}O_2$	18:0	7.48
Arachdic	eicosenoic	$C_{20}H_{38}O_2$	20:1	-
Behenic	Docosanoic	$C_{22}H_{44}O_2$	22:0	4.25

Table 4-2: Free fatty acid (FFA) profile of *Moringa oleifera* with other vegetable oils

Fatty acid	Moringa oleifera	Palm[a]	Rapeseed[a]	Soybean[a]	Sunflower
C16: 0	7.59	44.1	3.6	11	6.4
C18: 0	7.48	4.4	1.5	4	4.5
C18: 1	72.67	39.0	61.6	23.4	24.9
C12: 2	-	10.6	21.7	53.2	63.8
C18: 3	-	0.3	9.6	7.8	-[b]
C20: 0	-	0.2	-	-	-
C20: 1[c]	-	-	1.4	-	-
C22: 0	4.25	-	-	-	-
Other	-	1.1% C14: 0 traces of others	0.2% C22: 1	Traces	Traces

[a] Data from Gunstone and Harwood, 2007. These values constitute averages of numerous samples
[b] This may indicate traces (< 1%) or absence of these fatty acids.
[c]Eicosenoic acid.

4.4 Physical Properties Test and Analysis

The properties of the *Moringa oleifera* methyl ester (MOME) are summarised in Table 4.3 together with the relevant specifications from the biodiesel standards ASTM D6751 and EN 14214. It is shown that the values from the experiment data are within the standards.

Table 4-3: Physical properties analysis

Analysis	**Experiment**	**ASTM D6751 Standard**	**EN 14214 Standard**
Kinematic viscosity (mm^2/s)	4.93	1.9 – 6.0	3.5 – 5.0
Density (kg/m^3) at 15°C	872.7	870 – 900	860 – 900
Cloud point, °C	10.7	-	-
Pour point, °C	6.1	-	-
Cetane number	64.75	> 47	> 51

4.5 The tests for Moringa oleifera methyl ester

4.5.1Kinematic Viscosity

The term "kinematic" means that the measurement is made while fluid is flowing under the force of gravity. It is determined by measuring the time it takes for a volume of fluid to flow under gravity through a calibrated glass capillary viscometer. The kinematic viscosity is an important property regarding fuel atomisation and distribution. The

higher is the viscosity, the greater is the tendency of the fuel to form engine deposits. In this research, the kinematic viscosity of biodiesel compounds was determined at 40 °C, as this is the temperature prescribed in biodiesel standards according to ASTM D6751 standard method.

To determine the kinematic viscosity, different sizes of capillary tubes need to be prepared to support fluids of varying viscosity. In this research, viscosity number of No. 200 was selected as it is depending on the ASTM Standard. The equipment used was Cole – Parmer, USA. A sample of 30 mL biodiesel was pumped into the capillary tube until it reached the 'start mark'. The time taken for the oil to flow through the capillary tube can be converted directly to a kinematic viscosity using a simple calibration constant provided for each tube. The experiment was repeated for three times to get the accurate value.

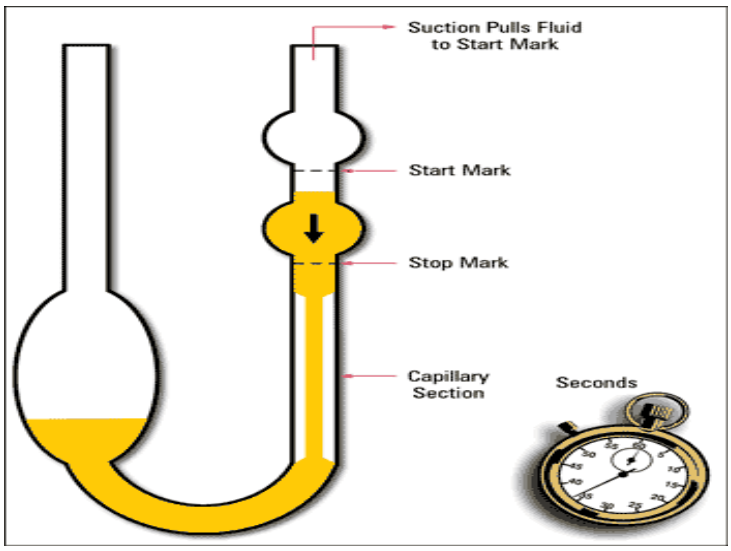

Figure 4-1: Capillary U-Tube Viscometer

4.5.2Density (ρ) Test

To determine the density of MOME produced, the equipment used was the Density Tester. The biodiesel sample needed was 20 mL and the chemical used was ethanol with 99 % of purity. Several steps are needed to complete the test:

1) Set the temperature of density meter to 15°C, and leave it until stabilized.

2) The inner tube of the density tester was cleaned by suction of ethanol into the tube. Repeat few times for this step to remove excess impurities and other mixing solutions.
3) Pumped the biodiesel sample into the density tester until it fully occupied the inner tube. This is to ensure no bubbles inside the tube and to avoid inaccurate reading.
4) Repeat step 3 for three times to get the average value.

4.5.3Cloud Point and Pour Point

The temperature at which oil starts to solidity is known as the cloud point. The cloud point is determined by visually inspecting for a haze to become visible as the fuel is cooled. In order to avoid waxing of the fuel, heating will be necessary while operating an engine at temperature below oil's cloud point. Using a product below its cloud point, may reduce the lubricating properties and may plug filters.

The sample of 20 mL biodiesel was poured into the test tube until it reached the level indicated on the test tube. Then, the sample was placed in the refrigerator. The sample had been observed every one minute until solid-crystals appeared. The temperature at which first observation of solid-crystals appeared was the cloud point at 10.7 °C.

After solid-crystals appeared, the sample was placed in a refrigerator. Once the temperature decreased and the sample still flowing inside the test tube, that temperature was considered as the pour point of at 6.1 °C.

4.5.4Cetane Number (CN)

One of the most commonly cited indicators of biodiesel fuel quality is *cetane* number. It measures the readiness of the fuel to auto-ignite when injected into the engine. Generally, it is dependent on the composition of the methyl ester oil and can impact the engine's start ability, noise level, and exhaust emissions. The *cetane* number of biodiesel is generally observed to be quite high. It is depends on the distribution of fatty acids in the original oil or fat from which it was produced. The longer the fatty acid carbon chains and the more saturated the molecules, the higher the *cetane* number.

In order to determine *cetane* number, 20 mL of MOME oil was needed. The oil was poured slowly into the *cetane* tester until it indicated label 'S' which means *cetane*